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LETTER DATED 30 MAY 1991 FROM THE DEPUTY PERMANENT REPRESENTATIVE OF NORWAY TO THE DEPUTY SECRETARY-GENERAL OF THE CONFERENCE ON DISARMAMENT TRANSMITTING A DOCUMENT ENTITLED, "VERIFICATION OF ALLEGED USE OF CHEMICAL WARFARE AGENTS: APPLICATION OF PROCEDURES AFTER A SIMULATED CHEMICAL ATTACK ON AN AIR BASE"

I have the honour to enclose a document entitled, "Verification of alleged use of chemical warfare agents: application of procedures after a simulated chemical attack on an air base".

I would be grateful if this document could be issued as an official document of the Conference on Disarmament as well as in the series of Working Papers for the Ad Hoc Committee on Chemical Weapons.

(Signed) Torbjørn Aalbu
Minister
Deputy Permanent Representative
to the Conference on Disarmament

GE.91-61020

VERIFICATION OF ALLEGED USE OF CHEMICAL WARFARE AGENTS

APPLICATION OF PROCEDURES AFTER A SIMULATED CHEMICAL ATTACK ON AN AIR BASE

Introduction

An extensive study of the possibility of verifying alleged use of chemical weapons was initiated by the Norwegian Ministry of Foreign Affairs in 1981. The research has been carried out by the Division for Environmental Toxicology of the Norwegian Defence Research Establishment at Kjeller near Oslo. The annual reports and working papers submitted to the Conference on Disarmament have been compiled in the publication Contributions by Norway to the conference on Disarmament 1982-1987 (document CD/813 dated 7 March 1988).

The 1988 report was mainly concerned with the development of complete procedures for verification of alleged use of chemical weapons, (CD/857 of 12 August 1988 and CD/861 of 22 August 1988), while the research in 1989 focussed on the development and testing of headspace gas chromatography, a new supplementary technique (CD/936 of 21 July 1989 and CD/940 of 31 July 1989).

The sample preparation method developed by the Norwegian research programme is based on the adsorption of chemical warfare agents from aqueous solutions onto a polymer sorbent and their subsequent removal by an organic solvent. This technique, referred to as sorbent extraction, was optimized in the 1989-1990 programme in order to isolate as efficiently as possible chemical warfare agents and related compounds from different sample materials. The procedure has been made as general as possible for use in screening samples suspected of being contaminated with chemical warfare agents. These results and conclusions were summarized in document CD/1008 dated 26 June 1990, and the research report was presented to the Conference with document CD/1019 dated 23 July 1990.

Based on the work carried out during the Norwegian research programme, a complete procedure for the verification of alleged use of chemical weapons has been developed, including the construction of a mobile field laboratory. This laboratory, contained in a wooden case, holds all the equipment and chemicals needed to carry out sampling, sample preparation and preliminary analyses in the field. The most important parts of this verification procedure are location of the contaminated area, sample collection, field analysis, sample transportation, sample preparation and sample analysis.

When the Chemical Weapons Convention enters into force, the Technical Secretariat will receive the results from the laboratories which analyze samples taken during the inspection of an area where use of chemical weapons is alleged to have taken place. To allow correct conclusions to be drawn from the results provided, it is important that the history of the

samples has been properly recorded. All available information from the inspection of the contaminated area, sampling, sample preparation and transport should be recorded and passed on to the Secretariat. Most of this information should also be passed on to the analytical laboratories, to make it easier to select the best suited analytical method and to evaluate the reliability of the results. It is also important that the samples are sealed and secured to prevent tampering during transport.

During this research programme, the evaluation of verification procedures by field exercises has been emphasized, in order to reveal problems that would not occur during laboratory testing.

This year's report is divided into two parts. The main part is an application of the complete procedures for verification of alleged use of chemical weapons, developed during the Norwegian research programme. It deals with handling of samples from a simulated attack with chemical weapons on an air base. Samples were collected in the area in question and treated in accordance with the procedures. In addition to the samples collected after the simulated attack, some samples of soil, sand, water and protective clothing were spiked with chemical warfare agents in the laboratory, transported to the air base, where they were exposed to the prevailing weather, and then treated in the same manner as the samples collected.

A form has been drawn up on which the sampling team should enter all information acquired during the localization of the contaminated area, sample collection, sample preparation, field analyses and sample transport. A transport log has also been drawn up which would be filled in by the personnel in charge of each section of the transport chain in order to keep a complete record of the history of the samples.

The second part of the report describes further improvement of the procedures for handling samples. This includes testing methods for homogenization of samples, and a comprehensive testing of solvents for extraction of different chemical warfare agents from various sample materials.

The report confirms earlier results on the advantages of the sorbent extraction technique for the detection of chemical warfare agents, and describes further improvements of the sample preparation and handling procedures. The report is a concrete contribution by experts at the Norwegian Defence Research Establishment to the negotiations on a Chemical Weapons Convention closely related to the Protocol on Inspection Procedures (CD/1046, Appendix I, Addendum, Part IV: Procedures in cases of alleged use of Chemical Weapons) and the relevant General Assembly resolutions on measures to uphold the authorities of the 1925 Geneva Protocol, in particular resolution 45/57 C endorsing the proposals of the group of qualified experts concerning technical guidelines and procedures to guide the Secretary-General in the conduct of

timely and efficient investigation of the reports of use of chemical and bacteriological (biological) or toxin weapons (document A/44/561 dated 4 October 1989).

Field exercise

An attack on an air base with chemical warfare agents was simulated by exploding a bottle filled with 200 g of the nerve agent simulant dipropyleneglycol monomethyl ether (DPM) 1.5 m above the ground. After the explosion, all visual evidence was removed before a team from the Norwegian Defence Research Establishment was called upon to verify the alleged use of chemical warfare agents on the air base. This team was unaware of the exact site of the explosion, and was required to identify the contaminated area, carry out field analyses, collect samples and treat them according to the Norwegian procedure, and transport them to the laboratory.

Five hours after the explosion of the simulant the inspection team arrived at the air base, and was transported to the area where eyewitnesses had seen a suspicious explosion of an artillery shell. The inspection was carried out as it would have been after a real incident, and the inspectors used respirators and complete protective clothing throughout the localization and sample collection phases. An area of 100 m x 100 m pointed out by the eyewitnesses was first inspected visually by two members of the team in order to find any remnants from the explosion. This inspection took about 7 minutes, but gave no indication of where the explosion had taken place. The inspection team then used a chemical agent monitor (CAM) and detection paper to localize the possible contaminated area. After 20-25 minutes total search time, an area contaminated with a nerve agent simulant was located; another 10 minutes were used to define the extent of this area. Detection paper was used to confirm the observations made with the CAM. The contaminated area was found to be 15m x 10m.

Twenty samples were collected throughout this contaminated area. Most of the samples were, however, collected near the point which on the CAM showed the highest concentration of the DPM simulant. Samples of about 50 g of soil, sand or water were collected. Reference samples from outside the contaminated area were also collected. The samples were placed in glass bottles with airtight screw caps, coded by sample number and sealed with wax, and then transported back to the laboratory. No further analyses were carried out on these samples.

It is extremely important to record information on the samples collected, the sampling methods and sampling site to allow correct conclusions to be drawn from the results obtained by sample analysis. A three-page form has therefore been drawn up

for use by the sampling team during the inspection. This form is included as an annex to this paper. On the first page, information on the sampling should be entered, including the reason for sampling, an indication of the priority or importance of the samples, description of samples and sampling site, sampling methods and results of field analyses. On the second page, a sketch of the sampling site should be drawn, indicating topography, any bomb craters observed and wind direction. The positions of the sampling sites should also be indicated on this sketch. The third page should be filled in if information on the attack is available. Reports from eyewitnesses, casualties, the effect on vegetation and the weather conditions since the attack should be entered here.

In addition to samples from this simulated attack, samples of 50 g of soil, sand and water, and 0.05 g (20 cm^2) of protective clothing were spiked in the laboratory with 1 mg of each of the nerve agents GA, GB, VX, and the vesicants Lewisite (L) and mustard (H). These samples were placed in closed vessels and transported to the air base where the vessels were opened and the samples exposed to the prevailing weather for 20 hours. Some of these samples were then treated in the field according to the standard Norwegian procedure and the others transported back to the laboratory and used for evaluation of different sample preparation methods.

In order to have a first indication of which chemical was present, some of the samples spiked with real chemical warfare agents were analyzed in the field using thin layer chromatography (TLC). The agents GA, GB, VX and H were detected in all sample materials (soil, sand, water and protective clothing). As Lewisite is rapidly hydrolyzed in water, it could not be detected in the aqueous samples.

After exposure to the prevailing weather for 20 hours, three replicates of each of the spiked samples were washed with 50 ml water and passed through a combination of C_{18} cartridges (top) and NH_2 cartridges (bottom) using a hand operated vacuum system. The cartridges were pre-wetted with 1 ml methanol and 10 ml water before application of the samples. The cartridges were labelled, put in a sealed plastic bag and placed in a thermos flask of dry ice for transport to the laboratory. The rest of the spiked samples were transported untreated in closed containers back to the laboratory for further analyses; a military aircraft was used for the transport, and military regulations were followed. All relevant information on the samples and sample preparation was entered on the form previously described. One copy of the form was transported together with the samples, and one copy was retained by the sampling team. A transport log has further been drawn up to follow the samples during transportation. One log followed each parcel, and the date and time were entered by the personnel in charge of each stage of transportation from the sampling site to the destination. The maximum permissible temperature during transport was filled in by the sampling team for the benefit of the personnel in charge of transport.

A transport log is shown as an annex to this paper.

At the laboratory, the chemical compounds were eluted from the C_{18} and NH_2 cartridges and the eluates analyzed by gas chromatography. One portion of each of the untreated samples was also analyzed by headspace gas chromatography without pretreatment.

The results of the analyses of the spiked and weathered samples show that GA, GB, VX and H were found in all sample materials using the C_{18} sorbent extraction technique. The headspace method is not useful for low-volatile compounds like VX, but detected all the compounds except sarin (GB) in sand and tabun (GA) and mustard (H) in soil samples. Because lewisite (L) decomposes very rapidly in contact with water, this compound was not detected in any of the samples analyzed.

Laboratory experiments

When the samples are received at the laboratory after transport from the field, they are generally split into several sub-samples for analysis by several different methods. To be sure that the sub-samples contain equal amounts of the chemical compounds to be determined, it is important that the main samples are homogenized and split properly. A method of homogenization of solid samples before splitting into sub-samples has been developed using a shaking machine for dry samples and a mortar for wet samples. In these experiments, the sarin impurity diisopropyl methylphosphonate (DIPMP) was used as a model substance and added to sand, soil and glass spheres at several different concentrations. When the homogenized samples were split into ten equal sub-samples and analyzed, DIPMP was found in all samples with satisfactory standard deviations.

In addition, the organic solvents methanol, acetone, dichloromethane, tetrachloromethane, chloroform, isopropanol, diethyl ether, ethylacetate, hexane and the mixtures methanol:chloroform (1:1) and chloroform:diethyl ether (1:1) were tested to evaluate which one most efficiently extracts selected chemical warfare agents and related compounds from different sample materials. The chemical compounds tabun (GA), sarin (GB), soman (GD), VX, mustard gas (H), and methyl 1,2,2-trimethylpropyl methylphosphonate (MTMP) were tested on the materials sand, soil, paper and butyl rubber. This experiment showed that the highest recovery rates were obtained with the methanol/chloroform mixture. This mixture also showed the smallest variations (SD) among the sample materials. There were only small differences between the results obtained with other extraction solvents, except for hexane, which consistently gave the lowest extraction yields. One problem observed with the methanol/chloroform mixture was that phase separation occurred with some types of samples.

Conclusions from field experiment

To evaluate the complete verification procedure, including the usefulness of the sampling forms and transport log, an attack with chemical weapons on an air base was simulated. The sampling team used the chemical agent monitor (CAM) and detection paper to localize and define the contaminated area in about 30 minutes. The CAM proved especially useful for this purpose.

To allow correct conclusions to be drawn from the results of the sample analysis, it is extremely important to record information during location of a contaminated area, sample collection, sample preparation, field analysis and sample transport . A form has therefore been drawn up for use by the sampling team during the inspection. This should be completed with information about the reason for taking samples, a description of the sample site, sampling methods, weather conditions and results of the field analysis. If eyewitness accounts or other information on the chemical attack is available, this should be included, together with reports on the inspection of any casualties. One copy of the form should be sent to the laboratory together with the samples and one copy retained by the sampling team. A transport log has also been drawn up, and this should follow each parcel during transportation. The date and time should be entered in this log by personnel in charge of each section of the transport.

The field exercise showed that thin layer chromatography (TLC) can be used as a rapid method for screening samples of chemical warfare agents in the field close to the contaminated area. This method gives an initial indication of which chemical agent is present in the samples, and could make it easier to select the methods of sample preparation and analysis which give the most reliable results in the laboratory.

The analysis of the samples spiked with real chemical warfare agents showed that the sorbent extraction (C_{18}) method could detect all spiked agents in all sample materials. The greatest advantage of this method is that chemical warfare agents can be isolated from the sample materials in the field immediately after sample collection. This minimizes the loss of information due to decomposition and evaporation of the agents during transport to the laboratory. In addition, the sorbent extraction technique is easy to carry out and does not require large amounts of equipment and chemicals.

Conclusions from Laboratory experiments

It is important that the samples are properly homogenized before they are split into several sub-samples for analysis with different techniques. A method has been developed whereby dry samples such as sand are homogenized in a shaking machine for one minute before they are split into several sub-samples. Wet solid samples such as soil are homogenized in a mortar for three minutes. Using these methods, satisfactory standard deviations were obtained for the content of DIPMP in the sub-samples.

An evaluation of different solvents for extracting selected chemical warfare agents from the sample materials sand, soil, butyl rubber, and paper showed that a mixture of methanol (50%) and chloroform (50%) gave the highest extraction yields. A number of other solvents also gave high recovery rates.

SAMPLE DOCUMENTATION. SAMPLING.	Page 1 of 3
Items 1-11 refer to the sampling procedure.	
1. Name : Date : Time : Position:	
2. Reason for sampling :	
3. Sample priority : First Second Third	
4. Sample code number :	d
a	e
b	f
c	g
5. Description of sample :	d
a	e
b	f
c	g
6. Method of obtaining the sample(s) :	d
a	e
b	f
c	g
7. Results from field analysis (CAM, TLC) :	
8. Weather conditions :	
9. Description of sample site (topography, vegetation etc) :	
10. Location of sample site (map reference UTM) :	

SAMPLE DOCUMENTATION. SAMPLING. Page 2 of 3

11. Sketch of site , topography, bomb craters etc.
Use cross and sample code number to give sample site.
Please indicate North-South and wind direction.

SAMPLE DOCUMENTATION. ATTACK.	Page 3 of 3
Items 12-17 refer to observations concerning the attack.	
12. Date :	Time :
13. Weather conditions since the attack :	
14. Symptoms of casualties (humans/animals) :	
15. Effect on vegetation :	
16. Description of the attack method of delivery, smell, sound etc)	
17. Description of munitions found at the site :	

18. Remarks :

TRANSPORT LOG.

1. Sampling carried out by : _____ _____

   Confirmed by : _____ _____

   Date : _____ Time : _____ Parcel number : _____

   Total number of parcels in shipment : _____

   Temp should not exceed : _____°C Description of parcel :

2. Transport and storage information.

Date and time received	Place received	Means of transport	Temperature during transport	Name and position

3. Received by _____

   Institution _____ Date : _____ Time : _____